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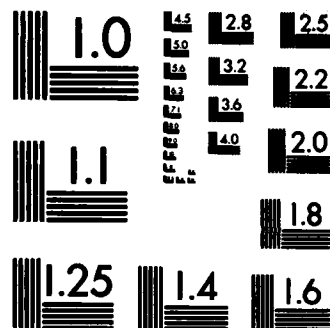
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SYNTHESES AND STRUCTURES OF ANIONIC CLOSO-RHODACARBORANE
CLUSTERS WHICH CONTAIN A FORMAL Rh(I) VERTEX

By

John A. Walker, Carolyn B. Knobler and M. Frederick Hawthorne*

Prepared for Publication

in

Journal of the American Chemical Society

Department of Chemistry and Biochemistry
University of California
Los Angeles, California 90024

March 15, 1983

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ABSTRACT

Isomeric rhodacarboranes $\text{closo-3,3-(Ph}_3\text{P)}_2\text{-3-H-3,1,2-RhC}_2\text{B}_9\text{H}_{11}$, 1a; $\text{closo-2,2-(Ph}_3\text{P)}_2\text{-2-H-2,1,7-RhC}_2\text{B}_9\text{H}_{11}$, 1b; and $\text{closo-2,2-(Ph}_3\text{P)}_2\text{-2-H-2,1,12-RhC}_2\text{B}_9\text{H}_{11}$, 1c, react with potassium tri-*sec*-butylborohydride in THF to produce anionic rhodacarboranes $[(\text{Ph}_3\text{P)}_2\text{-RhC}_2\text{B}_9\text{H}_{11}]^-$ (2a-c, respectively) tetraalkylammonium salts can be isolated in high yields.

The potassium 18-crown-6 salt (2a) and the tetramethylammonium salt (2b) were structurally characterized to determine whether polyhedral distortions could be detected in the solid structure. Complex 2a adopts a regular closo structure while 2b adopts a distorted polyhedral structure in which carbon atoms of the C_2B_3 bonding face are bent back into the cage, away from the metal. The metal vertex conformations of 2a and 2b with respect to the carborane ligand were in strict agreement with Mingos' molecular orbital predictions. Tetraethylammonium salts of 2a-c each reacted with CO (1 atm, 25° C) to produce in high yields $[\text{Et}_4\text{N}](\text{Ph}_3\text{P})(\text{CO})\text{RhC}_2\text{B}_9\text{H}_{11}]$ (3a-c, respectively). Similarly $[\text{Et}_4\text{N}]\text{2a}$ and $\text{K}[18\text{-crown-6}]\text{2b}$ reacted with ethylene (1 atm, 25° C) to produce the corresponding salts of $[(\text{Ph}_3\text{P})(\eta^2\text{-C}_2\text{H}_4)\text{RhC}_2\text{B}_9\text{H}_{11}]^-$ (4a and 4b, respectively). The new complexes reported here are quite reactive because of their anionic, five-coordinate nature.

Structural characterization of electron-rich metallocarboranes has been of interest since the earliest crystallographic studies showed that some late transition metal metallocarboranes adopt significantly distorted polyhedral structures.¹ The importance of the electronic configuration of the metal was immediately recognized², but more recent structural studies of 12-vertex platinum and palladium metallocarboranes have shown that the degree of polyhedral distortion is also a sensitive function of the ancillary ligands, the relative positions of the carbon atoms present in the carborane ligand and the metal itself.³ Prompted by these unique structural findings, Mingos has completed extended Hückel calculations on model icosahedral platinacarboranes and has explained the conformations and polyhedral distortions adopted by late transition metal metallocarboranes.^{3e,4} While there are many examples of icosahedral $d^8 L_2M(II)$ ($L=CO, RNC, PR_3$; $M=Ni, Pd, Pt$) metallocarboranes, isolation of isoelectronic $L_2M(I)$ ($M=Co, Rh, Ir$) metallocarboranes has proven to be more elusive. Only one example of the latter, namely, $3-(Ph_3P)-3-(CO)-4-(C_5H_5N)-3,1,2-RhC_2B_9H_{11}$, has been structurally characterized⁶ and the conformation of the metal vertex proved to be incompatible with molecular orbital predictions. Thus, in order to evaluate structural consequences of icosahedral d^8 rhodium (I) metallocarboranes we have synthesized and structurally characterized salts of $[3,3-(Ph_3P)_2-3,1,2-RhC_2B_9H_{11}]^{1-}$ and $[2,2-(Ph_3P)_2-2,1,7-RhC_2B_9H_{11}]^{1-}$. Structures of these two anions provide the first opportunity to evaluate the effects of introducing identical d^8 metal centers into isomeric 7,8- and 7,9- $C_2B_9H_{11}^{2-}$ ligands. Furthermore, we are not aware of any other structurally characterized monometallic anions which contain both formal Rh(I) and PPh_3 ligands.

Isomeric hydridorhodacarborane clusters $3,3-(Ph_3P)_2-3-H-3,1,2-RhC_2B_9H_{11}^{7a}$ (1a); $2,2-(Ph_3P)_2-2-H-2,1,7-RhC_2B_9H_{11}^{7a}$ (1b) and $2,2-(Ph_3P)_2-2-H-2,1,12-RhC_2B_9H_{11}^{7b}$ (1c) react with many bases (e.g., KOH, NaH, K-Selectride) at the rhodium vertex to generate air-sensitive anionic rhodacarboranes; 2a, 2b, 2c, respectively. Based on analytical and spectroscopic data,⁸ each of these anions was formulated as

$[(\text{Ph}_3\text{P})_2\text{RhC}_2\text{B}_9\text{H}_{11}]^{1-}$ available (85% yield) as crystalline tetraalkylammonium salts or potassium 18-crown-6 salts. Ethanol suspensions of complexes 2a-c reacted with dilute mineral acids to quantitatively regenerate the respective parent hydrido complexes 1a-c.

Dichloromethane solutions of complexes 2a-c were found to react readily with CO (1 atm, 25° C) to produce tetraethylammonium salts of $[(\text{Ph}_3\text{P})(\text{CO})\text{RhC}_2\text{B}_9\text{H}_{11}]^{1-}$ (3a-c, respectively) (80-95% yields).⁸ Similarly, 2a and 2b react with ethylene to produce (65-75% yield) the corresponding isomeric species $[(\text{Ph}_3\text{P})-(\eta^2\text{-C}_2\text{H}_4)\text{RhC}_2\text{B}_9\text{H}_{11}]^{1-}$ [4a, NEt_4 cation; 4b, K[18-crown-6]].^{8c} Complexes 3a-c were quite stable in the absence of air, but 4a and 4b slowly dissociated ethylene in solution.

To determine the nature and the extent of distortion produced by the electron-rich $(\text{Ph}_3\text{P})_2\text{Rh}$ (I) vertex in 2a and 2b, X-ray structure determinations were carried out on the potassium 18-crown-6 salt (2a)⁹ and the tetramethylammonium salt (2b).⁹

A projection of 2a is shown (Figure 1). Contrary to initial expectations of a slipped or distorted polyhedral structure, as in 3,3-(PEt_3)₂-3,1,2- $\text{PtC}_2\text{B}_9\text{H}_{11}^{3e}$, the rhodium vertex in 2a was found to be symmetrically bonded to the planar C_2B_3 face of the carborane ligand.

In accord with the molecular orbital predictions⁴, the P(1)-Rh-P(2) plane is nearly perpendicular to the mirror plane in the carborane ligand passing through B(8), the midpoint between C(1) and C(2), and B(10), the apical boron atom.

Bond distances and angles within the carborane framework are not unusual, overall icosahedral geometry closely resembles that found in 1a.¹⁰

The molecular structure of NMe_4^+ 2b is shown in Figure 2. Unlike 2a, the C_2B_3 bonding face of ion 2b shows significant deviation from planarity, with carbon atoms bent back into the cage, away from the metal. The non-planarity of this C_2B_3 face can be further described in terms of the angle between the normals to planes C(1)-B(6)-B(11)-C(7) and C(1)-B(3)-C(7), 15.7°. The P(1)-Rh-P(2) plane is nearly parallel to the mirror plane in the carborane ligand passing through B(3), the midpoint between

B(6) and B(11) and the apical boron atom B(9). This is the same metal vertex conformation found in $2,2-(\text{PPhMe}_2)_2-1,7-(\text{CH}_3)_2-2,1,7\text{-PtC}_2\text{B}_9\text{H}_{11}^{3c}$ and is calculated to be the preferred conformation.⁴

In the solid state, 2b adopts the metal conformation predicted by Mingos⁴; however, it is fluxional in solution. The variable temperature 81.02 MHz $^{31}\text{P}\{^1\text{H}\}$ NMR (d_6 -acetone) of 2b (room temperature) consists of a doublet centered at 51.7 ppm (downfield of external D_3PO_4) with a rhodium-phosphorus coupling constant of 212Hz. At 240K, this resonance collapses to a broad featureless peak centered at 52.5 ppm which at 190K sharpens into two separate doublets of doublets centered at 58.5 ppm ($J_{\text{Rh-P}} = 241\text{Hz}$. $J_{\text{P-P}} = 42\text{Hz}$.) and 44.3 ppm ($J_{\text{Rh-P}} = 186\text{Hz}$. $J_{\text{P-P}} = 42\text{Hz}$.). This behavior is consistent with hindered rotation about the metal vertex-carborane axis.¹¹ The low temperature limiting spectrum represents a fixed metal conformation which is probably the one observed in the crystal. The $^{31}\text{P}\{^1\text{H}\}$ NMR behavior of 2a and 2c show no temperature dependence; we assume that the barrier to rotation in these complexes is much smaller.

Qualitatively, polyhedral distortions of late transition metal metallacarboranes have been explained in terms of electron population of low lying anti-bonding orbitals involving the metal and the carborane ligand.⁴ This notion, coupled with the structures of 2a and 2b, suggests that the HOMO in 2b has greater anti-bonding character than the HOMO in 2a, precisely the conclusion derived from molecular orbital calculations. Similarly, one might expect complex 2c to adopt a structure similar to that found in $1,1-(t\text{-C}_4\text{H}_9\text{NC})_2-2-(\text{NMe}_3)-1,2\text{-PdCB}_{10}\text{H}_{10}^{3b}$ with the plane defined by the $(\text{Ph}_3\text{P})_2\text{Rh}$ fragment being perpendicular to the mirror plane of the carborane ligand and the carbon atom in the bonding face of the carborane ligand bent back into the cage away from rhodium. Verification awaits structural characterization of 2c.

The anionic, five-coordinate nature of the new complexes reported here has been exploited in a variety of ways. The accompanying communication describes novel cycloaddition reactions of aryl nitrile-N-oxides with the rhodium-carbonyl carbon bond

in complexes 3a and 3b.¹²

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FIGURE CAPTIONS

Figure 1. Anion of 2a.

Figure 2. Anion of 2b.

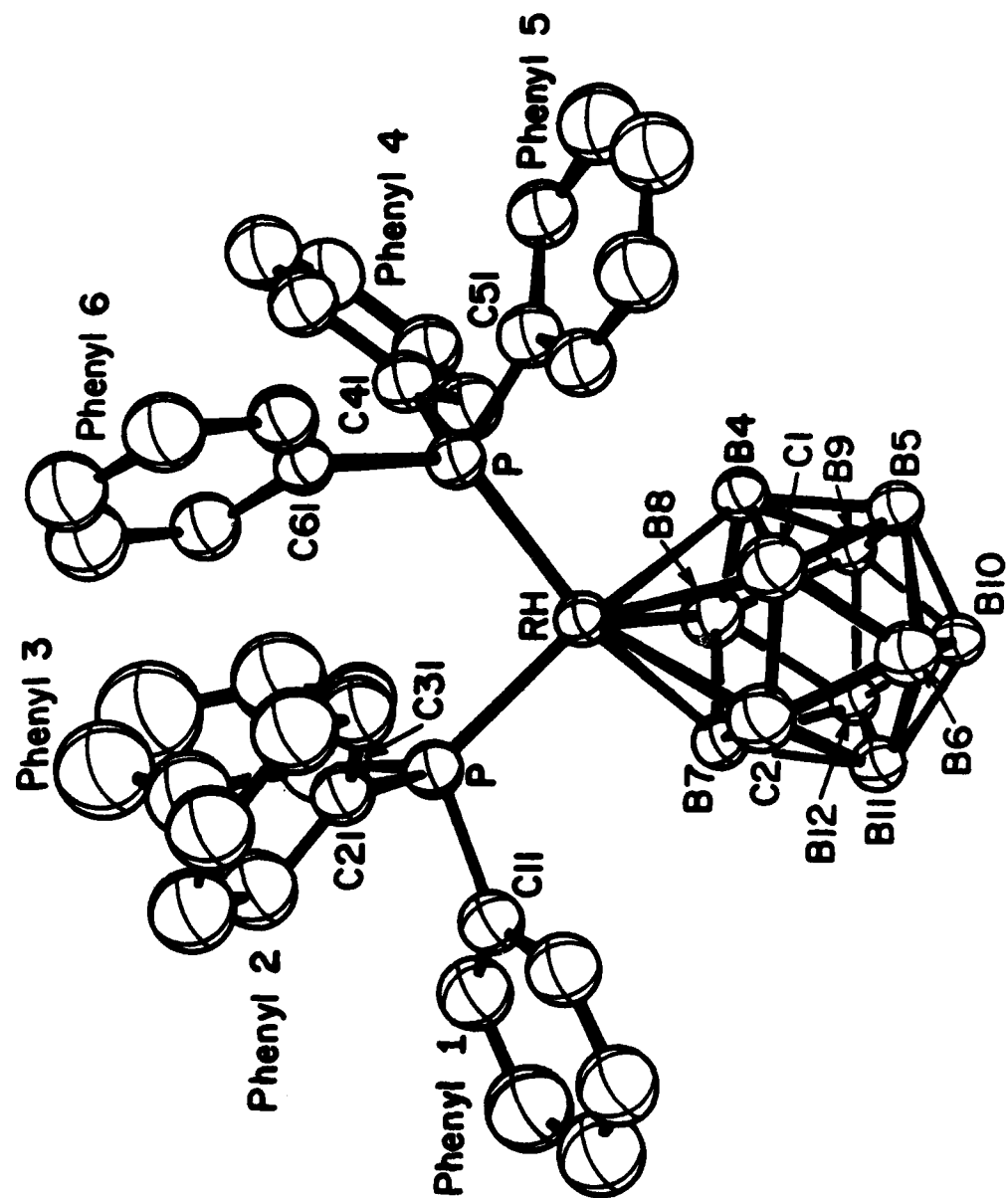


Figure 1.

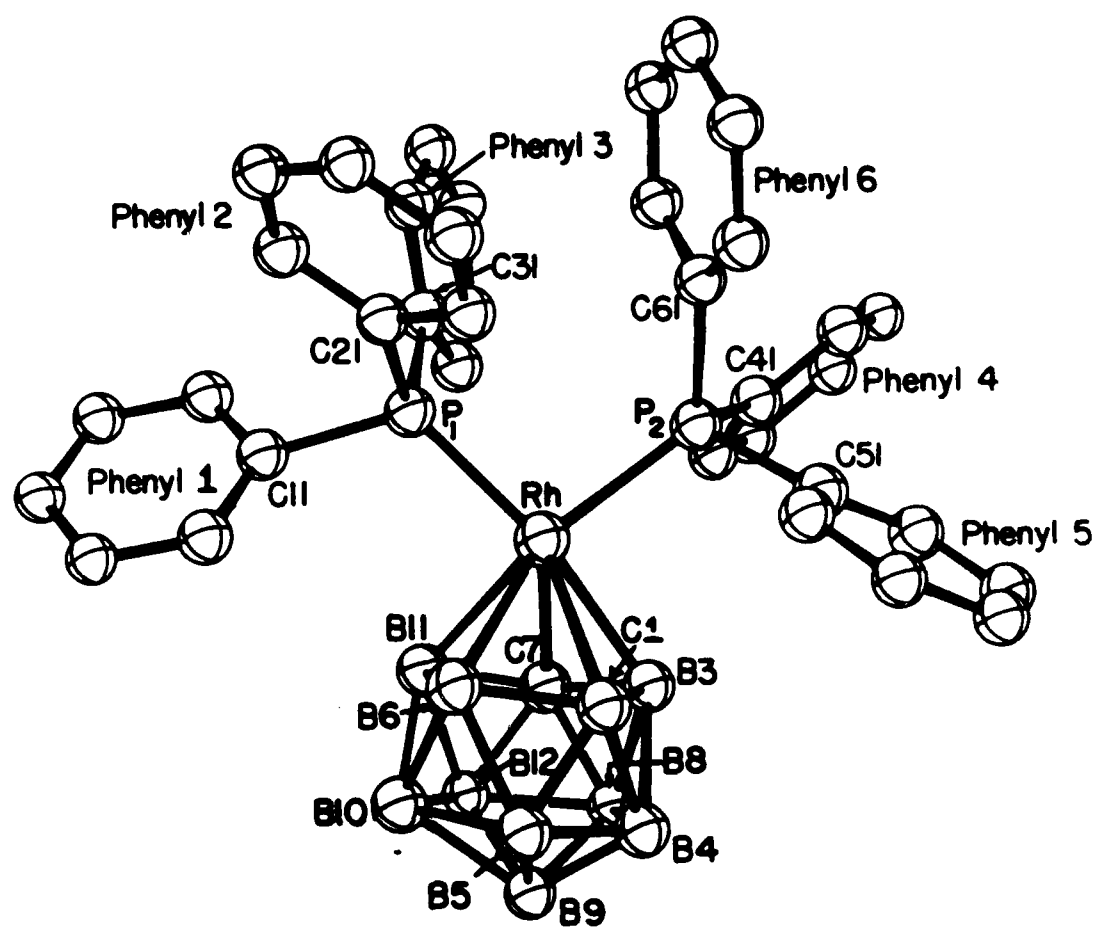


Figure 2.

SUPPLEMENTARY MATERIAL

All new compounds reported in this paper gave satisfactory analytical data for all elements except 2a whose precise composition was established by X-ray crystallography. Selected spectroscopic data for the new complexes are given below: 2a $^{31}\text{P}\{^1\text{H}\}$ NMR (referenced to external 85% D_3PO_4 , THF/ d_6 -benzene, 25° C), 47.3 ppm, (d, $J_{\text{Rh-P}} = 197\text{Hz}$). 2b; $^{31}\text{P}\{^1\text{H}\}$ NMR (d_6 -acetone, 25° C), 51.7 ppm (d, $J_{\text{Rh-P}} = 212\text{Hz}$). 2c; $^{31}\text{P}\{^1\text{H}\}$ NMR (d_6 -acetone, 25° C), 48.9 ppm (d, $J_{\text{Rh-P}} = 185\text{Hz}$)(b) 3a; $^{31}\text{P}\{^1\text{H}\}$ NMR (THF/ d_6 -benzene) 34.1 ppm (d, $J_{\text{Rh-P}} = 156\text{ Hz}$); $\text{IR}\nu_{\text{CO}} = 1920\text{ cm}^{-1}$ 3b; $^{31}\text{P}\{^1\text{H}\}$ NMR ($\text{CD}_2\text{Cl}_2/\text{CH}_2\text{Cl}_2$, 25° C) 42.7 ppm (d, $J_{\text{Rh-P}} = 171\text{Hz}$); IR, $\nu_{\text{CO}} = 1905\text{ cm}^{-1}$. 3c; $^{31}\text{P}\{^1\text{H}\}$ NMR ($\text{CD}_2\text{Cl}_2/\text{CH}_2\text{Cl}_2$, 25° C) 40.3 ppm (d, $J_{\text{Rh-P}} = 165\text{Hz}$); IR, $\nu_{\text{CO}} = 1920\text{ cm}^{-2}$. (c) 4a; $^{31}\text{P}\{^1\text{H}\}$ NMR (THF/ d_6 -benzene, 25° C), 48.0 ppm (d, $J_{\text{Rh-P}} = 183\text{Hz}$). 4b; $^{31}\text{P}\{^1\text{H}\}$ NMR (THF/ d_6 -benzene), 46.7 ppm (d, $J_{\text{Rh-P}} = 166\text{Hz}$).

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